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(54) FURNACE FOR A THERMOGRAVIMETRIC MICROBALANCE

(71) We, L. OERTLING LIMITED, a British Company, of Smethwick, Warley, Worcestershire, England, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to a furnace for a thermogravimetric microbalance. In the use of such microbalances a sample is continuously weighed as it is heated so that the effects of thermally-induced weight changes can be monitored. Weighing may be effected by balancing the sample against the same quantity of a reference substance unaffected by temperature, the reference substance being subjected to the same temperatures as the sample. Generally, an analysis requires the temperature to be elevated and maintained or varied in accordance with a predetermined programme. Before analysis of the next sample may begin it is necessary to allow the system to cool fully. This usually takes an appreciable time and restricts the use of the equipment. An object of the present invention is to alleviate this difficulty.

According to the invention there is provided a thermogravimetric microbalance comprising a balance beam from which a sample crucible is freely suspended in a furnace, which furnace comprises a housing defining a chamber, an electric heater in the chamber, the arrangement being such that the sample crucible may be freely suspended in the chamber from above to be heated by the heater, and means for liquid-cooling the housing.

The cooling liquid is usually water and the cooling means is conveniently constituted by ducts in the wall of the housing for carrying a flow of cooling water.

The heater may be a conventional electric element but preferably the heater is a self-supporting coil of metal sheathed mineral-insulated wire.

By "metal sheathed mineral insulated wire" is meant a wire comprising a metal capilliary tube along the inside of which runs a resistive heating wire, the wire being insulated from the tube by a packing of insulating mineral. A particularly useful form of metal sheathed mineral-insulated wire for use in accordance with the invention comprises a tube of Inconel ("Inconel" is a Trade Mark) about 1 mm. in diameter, an insulating packing within the tube of magnesium oxide, and an internal heating element within the magnesium oxide, and an internal heating element within the magnesium oxide packing comprising a nickel-chromium alloy wire.

The advantage of using a metal sheathed mineral-insulated coil of the kind described is that no former is required to support the coil. Therefore, the effective thermal mass of the coil is kept to a minimum. This shortens the thermal response time of the heating assembly. Furthermore, with a heater coil of this kind the metal tube end of the heating coil can readily make direct contact with the metal wall of the housing since there is no electrical insulation problem. This facilitates rapid cooling.

A small water-cooled furnace having a metal sheathed mineral-insulated heating coil of the kind described has a rapid thermal response and is particularly suitable for use in conjunction with a programmed controller for controlling the furnace temperature to follow a predetermined temperature program. Required temperature gradients and isothermal periods are accurately controllable. Preferably the furnace includes a temperature sensor within the heater coil for providing temperature feedback signals to the programmed controller. Preferably the temperature of the crucible is monitored by

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means of a further temperature sensor situated immediately beneath the crucible when in position.

Preferably, the thermogravimetric microbalance of the present invention includes two furnaces as described.

The invention will further be described with reference to the accompanying drawings, of which:—

Figure 1 is a schematic elevation of a thermogravimetric microbalance in accordance with the invention; and

Figure 2 is a cross-sectional elevation of a furnace shown in Figure 1.

Referring to Figure 1 the microbalance comprises a glass envelope 1 from which depend two stems 2 and 3. The stems fit into glass funnels 4 and 5 at the top of respective tubular glass columns 6 and 7. A balance beam 8 is pivoted at 9 and arm of the beam supports by means of a wire 10, 11, a respective crucible 12, 13. The crucibles are suspended in furnaces 14, 15 at the bottom of columns 6 and 7 respectively.

In use, the crucible 12 contains a sample to be analysed and the crucible 13 contains the same quantity of a reference substance. Both furnaces are heated together and equally in accordance with a predetermined temperature programme. The differential effect of temperature on the weights of the sample being analysed and the reference substance is indicated by the imbalance of the beam 8.

Both furnaces are the same but for the sake of illustration the furnace 14 is illustrated in Figure 2. The furnace comprises a tubular casing 16 of chromium-plated brass which defines a cavity 17 within which is a self-supporting heater coil 18 of metal sheathed mineral-insulated wire. The coil has a 1 mm. diameter capilliary tube of Inconel within which runs a nickel-chromium alloy heater wire. The heater wire is insulated from the tube by a packing of magnesium oxide powder.

The casing 16 has a series of water ducts D in which cooling water is circulated as to seal the water ducts. Thermal contact is made between the heater coil and the base shown by the arrows. A top cover 19 and a base 20 are fitted to the cylindrical part of the housing and 'O' ring seals 21 are provided 20 and this, in conjunction with the water cooling of the case, facilitates rapid cooling of the furnace.

The temperature of the furnace is controlled by a programmed controller (not shown) which supplies heating current to the coil to determine the temperature in accordance with a set programme. The temperature of the furnace is sensed by a temperature sensor 22 which may be a platinium or a thermocouple, output signals from the temperature sensor being applied as feedback signals to control the programmed controller. The metal sheathed mineral-insulated coil 18 has a particularly fast thermal response which allows accurate temperature control by the programmed controller.

The temperature of the crucible 12 and thus of the sample being analysed is given by the output from a thermocouple 23 supported just below the crucible.

An additional advantage of the arrangement described above with reference to the drawings is that weighing accuracy is enhanced. It is felt that this may be due in part to the low thermal mass of the furnace (a typical external diameter of the furnace being 50—100 mm.) and in part to the water cooling. Both of these factors affects the amount of heat which is allowed to rise from the furnace up the column of the microbalance.

WHAT WE CLAIM IS:—

1. A thermogravimetric microbalance comprising a balance beam from which a sample crucible is freely suspended in a furnace, which furnace comprises a housing defining a chamber, an electric heater in the chamber, the arrangement being such that the sample crucible may be freely suspended in the chamber from above to be heated by the heater, and means for liquid-cooling the housing.

2. A microbalance as claimed in Claim 1 wherein the cooling means comprises ducts in the wall of the furnace housing for carrying a flow of cooling liquid.

3. A microbalance as claimed in either of the preceding claims wherein the heater is a self-supporting coil of metal sheathed mineral-insulated wire (as hereinbefore defined).

4. A microbalance as claimed in Claim 3 wherein the heater comprises an outer metal tube, an insulating packing within the tube of magnesium oxide, and an internal heating element within the magnesium oxide packing comprising a nickel-chromium alloy wire.

5. A microbalance as claimed in Claim 3 or Claim 4 wherein the heating coil makes direct thermal contact with the base wall of the furnace housing.

6. A microbalance as claimed in any of Claims 3 to 5 wherein there is provided a temperature sensor within the heater coil.

7. A microbalance as claimed in any of the preceding claims wherein there is provided a temperature sensor in the furnace immediately beneath the sample crucible when in position.

8. A microbalance as claimed in any one of the preceding claims wherein the balance beam has a second crucible freely suspended

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therefrom in a second furnace as defined in any one of said claims.

9. A thermogravimetric microbalance substantially as hereinbefore described with reference to Figures 1 and 2 of the accompanying drawings.

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FIG.1.

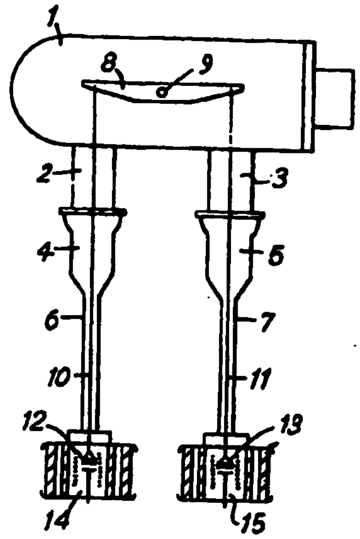
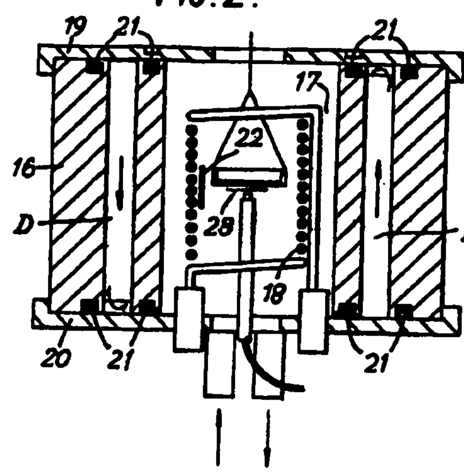


FIG.2.



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